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AN ENERGY-BEAM-CURABLE COMPOSITION USED FOR PRIMARY  
COATING OF AN OPTICAL FIBER

[Hikari faibaa ichiji hifukuyo kassei  
enerugiisen kohkayohgata soseibutsu]

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[There are no amendments to this patent.]

Claims

/2

1. An energy-beam-curable composition used as a primary coating for optical fibers composed of a compound having 1-4 oxetane rings and a photocationic polymerization initiator.

2. The energy-beam-curable composition used as a primary coating for optical fibers specified in Claim 1 above, wherein a compound containing an epoxy group also is included.

3. The energy-beam-curable composition used as a primary coating for optical fibers specified in Claim 1 or 2 above, wherein a compound containing a vinyl ether group is also included.

4. The energy-beam-curable composition used as a primary coating for optical fibers specified in Claim 1, 2 or 3 above, wherein a compound containing a (meth)acryloyl group and a photoradical polymerization initiator also are included.

Detailed explanation of the invention

[0001]

## Industrial application field

The present invention pertains to an energy-beam-curable composition used as a primary coating for optical fibers made of a compound containing an oxetane ring and which is used in the production of optical fibers as well as in the field of information communication. Also, in the specification of the present invention, an acryloyl group or a methacryloyl group is referred to as a (meth)acryloyl group.

[0002]

## Prior art

It is well known that optical fibers are very brittle and are likely to be scratched, and furthermore, optical transmission loss is increased by contamination. For this reason, a primary coating of a material having a low modulus of elasticity is applied to the surface of the glass right after production of the optical fiber, and an additional further secondary coating of a material having a high modulus of elasticity has been applied in the past. As a material used for primary coating of the above-mentioned optical fiber, a composition capable of undergoing curing upon application of an activating energy beam has been used in recent years because of high productivity. For the active energy-beam-curable composition used for the above-mentioned

application, a composition comprising an unsaturated polyurethane capable of curing by a radical polymerization reaction initiated by an activating energy beam, in specific terms, a composition comprising a polyether urethane acrylate, is widely used. For examples of the polyether polyol component used as the raw material for the polyether urethane acrylate in this case, polyethylene glycol, polypropylene glycol, polybutylene glycol, polytetramethylene glycol, copolymers of propylene oxide and ethylene oxide, copolymers of tetrahydrofuran and propylene oxide, alkylene oxide adducts of bisphenol A, etc., can be mentioned. Among those listed above, polyether urethane acrylates, in which polyethylene glycol is used as the raw material, are widely used, but the resistance to hydrolysis and resistance to water absorption of compositions made with the above-mentioned polyether urethane acrylate are inadequate; thus, polyether urethane acrylates made with raw materials made of polypropylene glycol or polytetramethylene glycol which have excellent properties have been widely used in recent years. Despite low viscosity and excellent handling properties, the curing characteristics of a composition made of polyether urethane acrylate produced using polypropylene glycol are inadequate and the heat resistance of the cured film is inadequate. On the other hand, the curing characteristics of compositions made of polyether urethane acrylates produced using polytetramethylene glycol are good and the heat resistance of the cured film is good, but the crystallinity is high and the material remains solid at ambient temperature. Thus, the handling properties are poor, and because of the high tensile modulus of elasticity of the cured film produced, it is necessary to add a reactive diluent, such as a (meth)acrylate with a low molecular

weight, but addition of the reactive diluent leads to a problem of reduced heat resistance of cured films formed from the composition. In an attempt to eliminate the above-mentioned problems, a composition made of a polyether urethane acrylate in which a copolymer of tetrahydrofuran and propylene oxide is used as the polyether polyol component is being studied, but curing properties, heat resistance, etc., remain inadequate.

[0003]

As activating energy-beam curing technology other than an activating energy-beam-initiated radical polymerization reaction, activating energy-beam-initiated cationic polymerization technology is used in practice. Since the activating energy-beam-initiated cationic polymerization is not inhibited by oxygen, it is not necessary for the reaction to be carried out in an inert atmosphere, and the polymerization reaction can be done rapidly and completely in air, which is an advantage. Until now, the activating energy-beam-initiated cationic polymerization technology has been used mainly for polymerization of two types of monomers comprising epoxy resins and vinyl ethers. In particular, photocurable epoxy resins have excellent adhesion and the cured films exhibit high heat resistance and chemical resistance. However, the photopolymerization reaction rate is relatively low for the majority of conventional photocurable epoxy resins, and production efficiency is a problem. On the other hand, in many cases, photocurable vinyl ethers are volatile and have a strong odor. Furthermore, compared with photocurable epoxies, shrinkage during curing is more obvious and adhesion is

poor; in addition, resistance to absorption of water and resistance to hydrolysis remain inadequate.

[0004]

Problems to be solved by the invention

The inventors carried out much research in an effort to produce an activating energy-beam-curable composition that can be used effectively for the primary coating of optical fibers and which is capable of eliminating the above-mentioned problems and has excellent curing properties, provides a cured film with long-lasting durability, and has properties such as high heat resistance, resistance to hydrolysis, and resistance to absorption of water, as well as a low tensile modulus of elasticity.

[0005]

Means to solve the problems

As a result of much research carried out by the inventors, they discovered that a composition made of a cyclic ether having a specific structure is capable of eliminating the above-mentioned problems of the existing technology, and the result is the present invention. Thus, the first invention of the present application is an energy-beam-curable composition used as a primary coating for optical fibers which comprises a compound having 1-4 oxetane rings and a photocationic polymerization initiator. The second invention is the energy-beam-curable

composition used as a primary coating for optical fibers specified in the first invention, wherein a compound containing an epoxy group also is included. The third invention is the energy-beam-curable composition used as a primary coating for optical fibers specified in the first invention or second invention, wherein a compound containing a vinyl ether group also is included. The fourth invention is the energy-beam-curable composition used as a primary coating for optical fibers specified in the first invention, the second invention, or the third invention, wherein a compound containing a (meth)acryloyl group and a photoradical polymerization initiator are also included. In the following, the present invention is explained in further detail. /3

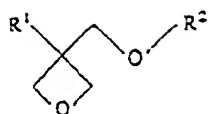
[0006]

○ Compound having 1-4 oxetane rings

The compound containing an oxetane ring used in the present invention includes 1-4 oxetane rings. When a compound containing 5 or more oxetane rings is used, the flexibility of the cured film formed by the composition is lost, and properties suitable for application as a primary coating for optical fibers cannot be obtained. Many different compounds can be used as the compound containing oxetane rings in the present invention as long as the compound includes 1-4 oxetane rings. As compounds containing 1 oxetane ring, compounds such as the one shown in general formula (1) below can be mentioned.

[0007]

[Structure 1]



(1)

[0008]

In general formula (1),  $R^1$  represents a hydrogen atom, an alkyl group with 1-6 carbon atoms such as methyl, ethyl, propyl, and butyl, a fluoroalkyl group with 1-6 carbon atoms, or an allyl, aryl, furyl, or thienyl group.  $R^2$  represents an alkyl group with 1-6 carbon atoms such as methyl, ethyl, propyl, and butyl, an alkenyl group with 2-6 carbon atoms such as 1-propenyl, 2-propenyl, 2-methyl-1-propenyl, 2-methyl-2-propenyl, 1-butenyl, 2-butenyl, or 3-butenyl, a group having an aromatic ring such as phenyl, benzyl, fluorobenzyl, methoxybenzyl, and phenoxyethyl group, an alkylcarbonyl group having 2-6 carbon atoms such as ethylcarbonyl, propylcarbonyl, or butylcarbonyl, an alkoxycarbonyl group with 2-6 carbon atoms such as ethoxycarbonyl, propoxycarbonyl, and butoxycarbonyl, an N-alkylcarbamoyl group with 2-6 carbon atoms such as ethylcarbamoyl, propylcarbamoyl, butylcarbamoyl, or pentylcarbamoyl, etc.

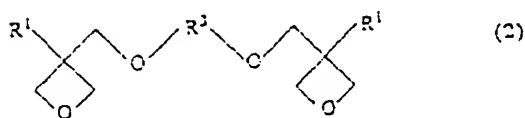


[0009]

Also, for compounds having 2 oxetane rings, a compound such as the one shown in the following general formula (2) can be mentioned.

[0010]

[Structure 2]

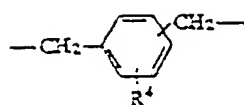


[0011]

In [general] formula (2),  $R^1$  represents groups such as those listed for the above-mentioned general formula (1).  $R^2$  represents, for example, a linear or branched alkylene group such as ethylene, propylene, and butylene, a linear or branched polyoxyalkylene group such as polyoxyethylene and polyoxypropylene, a linear or branched unsaturated hydrocarbon group such as propenylene, methylpropenylene, and butenylene, a carbonyl group and an alkylene group that includes carbonyl, an alkylene group that includes a carboxy group or an alkylene group that includes carbamoyl, etc. Furthermore,  $R^3$  is a polyvalent group selected from among the groups shown in [general] formulas (3), (4), and (5) below.

[0012]

[Structure 3]



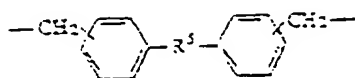
(3)

[0013]

In [general] formula (3),  $R^4$  represents a hydrogen atom, an alkyl group with 1-4 carbon atoms such as methyl, ethyl, propyl, and butyl, an alkoxy group with 1-4 carbon atoms such as methoxy, ethoxy, propoxy and butoxy, a halogen atom such as chlorine and bromine, nitro group, cyano group, mercapto group, lower alkylcarboxyl group, carboxy group, or carbamonyl group.

[0014]

[Structure 4]



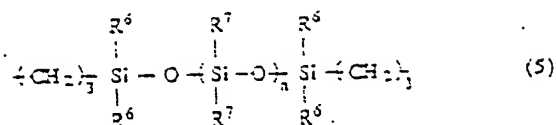
(4)

[0015]

In [general] formula (4),  $R^5$  represents an oxygen atom, a sulfur atom, methylene group, NH, SO, SO<sub>2</sub>, C(CF<sub>3</sub>)<sub>2</sub>, or C(CH<sub>3</sub>)<sub>2</sub>.

[0016]

[Structure 5]

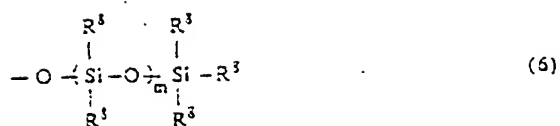


[0017]

In [general] formula (5),  $R^6$  represents an alkyl group or aryl group with 1-4 carbon atoms such as methyl, ethyl, propyl, and butyl.  $n$  is an integer in the range of 0-2000.  $R^7$  represents an alkyl group or aryl group with 1-4 carbon atoms such as methyl, ethyl, propyl, and butyl.  $R^7$  could also be a group selected from among the groups shown in [general] formula (6) below.

[0013]

[Structure 6]

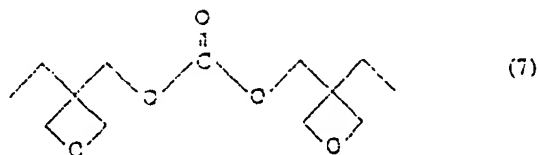


[0019]

In [general] formula (6),  $\text{R}^3$  represents an alkyl group or aryl group with 1-4 carbon atoms such as methyl, ethyl, propyl, and butyl.  $m$  is an integer in the range of 0-100. For specific examples of a compound having 2 oxetane rings, compounds such as those shown in [general] formulas (7) and (8) below can be mentioned.

[0020]

[Structure 7]

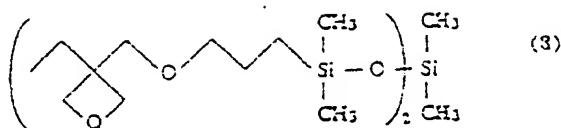


[0021]

The compound shown in [general] formula (7) is a compound having an ethyl group for  $R^1$  a carboxy group for  $R^3$  in [general] formula (2).

[0022]

[Structure 8]



[0023]

The compound shown in [general] formula (8) is a compound having an ethyl group for  $R^1$  in general formula (2) and a methyl group in which  $R^3$  is the same as  $R^5$  and  $R^7$ , and  $n$  is 1 in general formula (5).

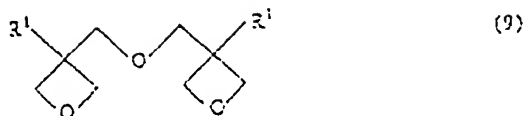
[0024]

In a compound having 2 oxetane rings, as a suitable example other than the compounds shown above, the compound shown in general formula (9) can be mentioned. In [general] formula (9),

$R^1$  represents the same groups as those shown in general formula (1) above.

[0025]

[Structure 9]

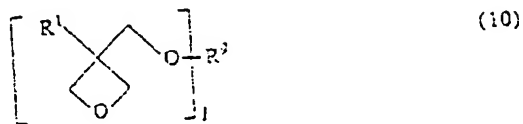


[0026]

For a compound having 3-4 oxetane rings, compounds such as those shown in general formula (10) below can be mentioned.

[0027]

[Structure 10]



[0028]

In [general] formula (10),  $R^1$  represents the same groups as those shown in the above-mentioned general formula (1).  $R^9$  represents, for example, a branched alkylene group with 1-12 carbon atoms such as those shown in [general] formulas (11)-(13) below, branched polyoxyalkylene group such as those shown in [general] formula (14) below, or a branched polysiloxane group such as those shown in [general] formula (15) below.  $j$  is either 3 or 4.

/5

[0029]

[Structure 11]

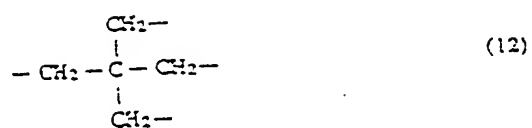


[0030]

(In [general] formula (11),  $R^{10}$  represents a lower alkyl group such as methyl, ethyl, or propyl.)

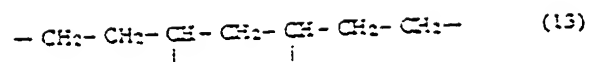
[0031]

[Structure 12]



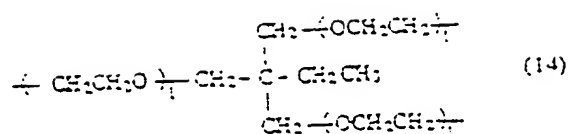
[0032]

[Structure 13]



[0033]

[Structure 14]



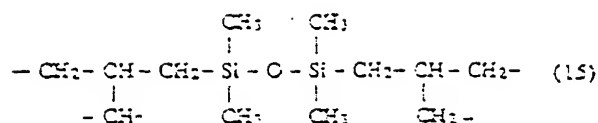


[0034]

(In [general] formula (14),  $l$  is an integer in the range of 1-10.)

[0035]

[Structure 15]

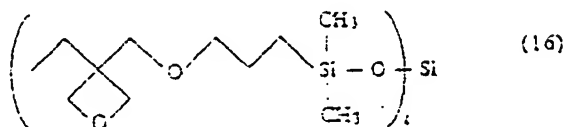


[0036]

For a compound having 3-4 oxetane rings, compounds such as those shown in the following general formula (16) can be mentioned.

[0037]

[Structure 16]

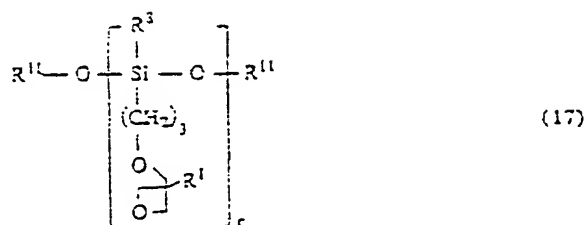


[0038]

Furthermore, as an example of a compound having 1-4 oxetane rings other than those shown above, a compound shown in [general] formula (17) below can be mentioned.

[0039]

[Structure 17]



[0040]

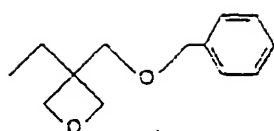
In [general] formula (17),  $\text{R}^3$  represents the same groups as those shown in [general] formula (6) above.  $\text{R}^{\text{II}}$  represents an alkyl group with 1-4 carbon atoms such as methyl, ethyl, propyl, or butyl or a trialkyl silyl group, and  $r$  is 1-4.

[0041]

As desirable specific examples of oxetane compounds that can be used in the present invention, compounds such as those shown below can be mentioned.

[0042]

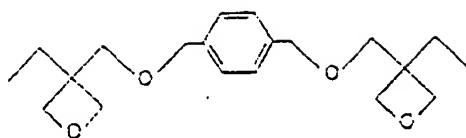
[Structure 18]



(18)

[0043]

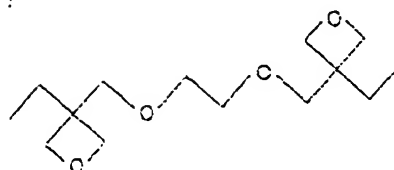
[Structure 19]



(19)

[0044]

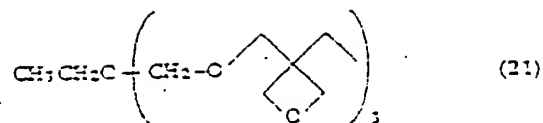
[Structure 20]



(20)

[0045]

[Structure 21]

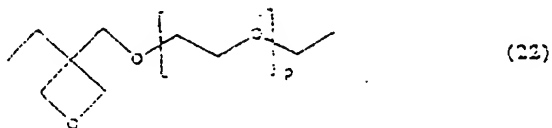


[0046]

Furthermore, in addition to those listed above, compounds having 1-4 oxetane rings and having a molecular weight of approximately 1000-5000 can be mentioned. For the above-mentioned examples, compounds such as those shown below can be mentioned.

[0047]

[Structure 22]

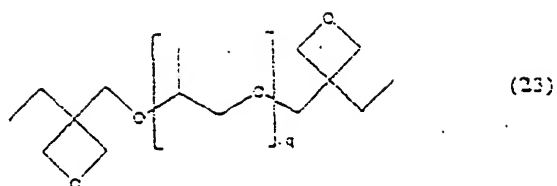


[0048]

In this case, p is 20-200.

[0049]

[Structure 23]

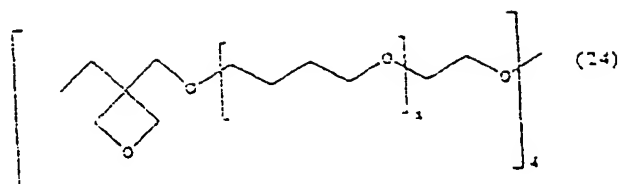


[0050]

In this case, q is 15-100.

[0051]

[Structure 24]



[0052]

In this case, s is 20-200.

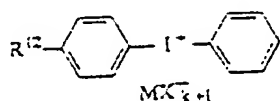
[0053]

# O Photocationic polymerization initiator

Many different types of photocationic polymerization initiators can be used in the composition of the present invention. As a suitable example of initiators used in this case, diallyliodonium salts and triarylsulfonium salts can be mentioned. Typical examples of photocationic polymerization initiators are shown below.

[0054]

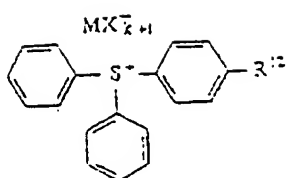
[Structure 25]



(25)

[0055]

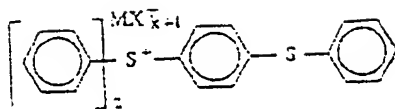
[Structure 26]



(26)

[0056]

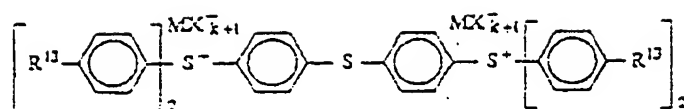
[Structure 27]



(27)

[0057]

[Structure 23]



(23)

[0058]

[In the formula,]  $\text{R}^{12}$  represents a hydrogen atom or an alkyl group with 1-13 carbon atoms or an alkoxy group with 1-13 carbon atoms,  $\text{R}^{13}$  represents a hydrogen atom, hydroxyalkyl group, or hydroxyalkoxy group, and hydroxyethoxy group is especially desirable. M is a metal and is preferably antimony; X represents fluorine or another halogen; and k is the valence of the metal, for example, in the case of antimony, it is 5. The mixing ratio of the photocationic polymerization initiator is preferably in the range of 0.1-20 wt% for the compound having an oxetane ring or for the total amount of said compound and the compound containing an epoxy group and/or the compound containing a vinyl ether group, when included, and a value in the range of 0.1-10 wt% is especially desirable. When the mixing ratio is below 0.1 wt%, the curing properties are inadequate; when the mixing ratio exceeds 20 wt%, the light transmittance is inadequate, uniform curing cannot be achieved, and the flatness



of the surface of the coated film cannot be achieved in some cases.

[0059]

Furthermore, in each of the chemical formulas showing a compound having 1-4 oxetane rings or a photocationic polymerization initiator, the groups indicated by the same codes in a single molecule may be the same or different.

[0060]

#### ○ Other compounds

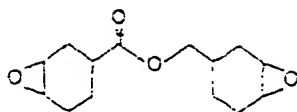
In addition to the above-mentioned essential compounds, other components can be included in the composition of the present invention. The second claim of the present invention is an energy-beam-curable composition used as a primary coating for optical fibers, wherein a compound having an epoxy group also is included in the composition of the Claim 1 of the present invention. In this case, when the compound having an epoxy group is included in the composition, the curing rate of the composition can be further improved. For the compound having an epoxy group, many different types can be used. For example, an epoxy compound having one epoxy group, phenyl glycidyl ether, and butyl glycidyl ether can be mentioned, and for epoxy compounds having two or more epoxy groups, hexanediol diglycidyl ether, tetraethylene glycol diglycidyl ether, trimethylolpropane triglycidyl ether, bisphenol A diglycidyl ether, novolak epoxy compounds, etc., can be mentioned. It is especially desirable to

use an alicyclic compound in the present invention. For example, the compounds shown below can be mentioned.

[0061]

[Structure 29]

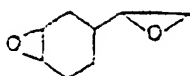
/3



(29)

[0062]

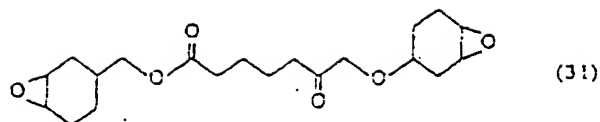
[Structure 30]



(30)

[0063]

[Structure 31]



[0064]

In this case, as a mixing ratio for the compound having the epoxy groups, 5-95 parts by weight are suitable for 100 parts by weight of the total amount of the compound having 1-4 oxetane rings and the compound having the epoxy groups.

[0065]

Claim 3 of the present invention is an energy-beam-curable composition used as a primary coating for optical fibers, wherein a compound having a vinyl ether group is included in the composition of Claim 1 or 2 of the present invention. In this case, when a compound having a vinyl ether group is included in the composition, the curing rate of the composition can be improved further. For compounds containing a vinyl ether group, many different ones can be used. For example, for compounds having 1 vinyl ether group, hydroxyethyl vinyl ether, hydroxybutyl vinyl ether, dodecyl vinyl ether, propenyl ether propylene carbonate, cyclohexyl vinyl ether, etc., can be mentioned. For compounds having two or more vinyl ether groups, cyclohexanedimethanol vinyl ether, triethylene glycol divinyl

ether, novolak divinyl ether, etc., can be mentioned. In this case, for an example of the mixing ratio of the compound having vinyl ether groups, 5-95 parts by weight are suitable for 100 parts by weight of the total amount of the compound having 1-4 oxetane rings and the compound having a vinyl ether group.

[0066]

Claim 4 of the present invention is an energy-beam-curable composition used as a primary coating for optical fibers wherein a compound having a (meth)acryloyl group and a photoradical polymerization initiator are included in the composition of Claim 1, 2, or 3 of the present invention. In this case, when the compound containing a (meth)acryloyl group is included in the composition, further adjustment of the viscosity of the composition and improvement in the hardness of the coated film formed by the composition can be achieved. For compounds having a (meth)acryloyl group, many different ones can be used. For example, for compounds containing one (meth)acryloyl group, (meth)acrylate phenol, nonylphenol, and 2-ethylhexanol, (meth)acrylates of alkylene oxide adducts of alcohols thereof, etc., can be mentioned. For compounds having two (meth)acryloyl groups, di(meth)acrylates of bisphenol A, isocyanuric acid, ethylene glycol, and propylene glycol, di(meth)acrylates of alkylene oxide adducts of alcohols of thereof, etc., can be mentioned. For a compound having three (meth)acryloyl groups, tri(meth)acrylates of pentaerythritol, trimethylolpropane, and isocyanuric acid, tri(meth)acrylates of alkylene oxide adducts of alcohols of thereof, etc., can be mentioned, and for a compound having four or more (meth)acryloyl groups, poly(meth)acrylates of

pentaerythritol, dipentaerythritol, etc., can be mentioned. Furthermore, conventional acrylic monomer oligomers such as urethane acrylate having a urethane bond as the principle chain, polyester acrylate having an ester bond as the principle chain, and epoxy (meth)acrylate, in which an acrylic acid is added to an epoxy compound, can be mentioned. In this case, for the mixing ratio of the compound having a (meth)acryloyl group, 5-95 parts by weight are suitable for 100 parts by weight of the total amount of the compound having 1-4 oxetane rings and the compound having a (meth)acryloyl group. In Claim 4 of the present invention, a photocationic polymerization initiator is added to the composition. Many different types of photocationic polymerization initiators can be used, and as suitable examples, benzophenones and derivatives thereof, benzoin alkyl ether, 2-methyl-[4-(methylthio)phenyl]-2-morpholino-1-propanone, benzyl dimethyl ketal, 1-hydroxycyclohexyl phenyl ketone, 2-hydroxy-2-methyl-1-phenylpropan-1-one, alkylphenyl glyoxylate, diethoxyacetophenone, 2-benzyl-2-dimethylamino-1-(4-morpholinophenyl)-1-butanone, and acylphosphine oxide, etc., can be mentioned. As for the mixing ratio of the above-mentioned photocationic polymerization initiators, 0.01-20 wt% for the compound containing (meth)acryloyl group is desirable.

[0067]

/3

Furthermore, in the present invention, one or more different compounds selected from the group consisting of the above-mentioned compounds containing epoxy groups, the compounds containing vinyl ether groups, and the compounds containing (meth)acryloyl groups can be added to the composition of the

first invention. In this case, it is desirable to add 5-95 parts by weight of the compound having 1-4 oxetane rings for 100 parts by weight of the total amount of the compound having 1-4 oxetane rings, the compound containing epoxy groups, the compound containing vinyl ether groups, and the compound containing (meth)acryloyl groups.

[0068]

The composition of the present invention can include inert components such as inorganic fillers, dyes, viscosity modifiers, treatment agents, and ultraviolet blocking agents in amounts of not more than 100 parts by weight for 100 parts by weight of the curable component.

[0069]

In addition to the photocationic polymerization initiator and/or photoradical polymerization initiator, a photosensitizer also can be included in the composition of the present invention, and adjustment of the wavelength in the UV region can be carried out. As a typical example of sensitizers that can be used in the present invention, those disclosed in Crivello (J.V. Crivello, Adv. in Polymer Sci., 62, 1(1984)) can be mentioned, and in specific terms, pyrene, perylene, Acrydine Orange, thiokanthone, 2-chlorothiokanthone, benzoflavin, etc., can be mentioned.

[0070]

# ○ Manufacturing method and application method

The manufacturing method of the composition of the present invention is not especially limited, and production can be carried out by stirring or mixing the essential components or essential components and other components that are added, as needed. The method of application of the composition of the present invention is not especially limited either, and conventional methods used for coating optical fibers can be used in this case as well. For example, a method in which coating of the composition of the present invention onto the surface of the optical fiber is carried out and an activating energy beam is subsequently applied to produce curing can be used. For the optical fiber onto which the composition of the present invention is to be applied, many different types can be used and the thickness of the composition can be appropriately selected according to the intended application. For the activating energy beam, ultraviolet (radiation), X-rays, electron beams, etc., can be mentioned. When ultraviolet is used for curing, many different types of light sources can be used, and for example, a pressurized or high-pressure mercury lamp, metal halide lamp, xenon lamp, electrodeless discharge lamp, carbon arc lamp, etc., can be mentioned. When an electron beam is used for curing, many different types of irradiation devices can be used, for example, Cockcroft-Walton, van de Graaff, or resonance potential transformer, etc., can be mentioned, and for the electron beam, an energy level in the range of 50-1000 eV is desirable, and in the range of 100-300 eV is especially desirable. Since it is

possible to use an inexpensive device, it is desirable to use ultraviolet for curing the composition.

[0071]

#### Application examples

In the following, application examples and comparative examples are described and the present invention is explained further in specific terms. It should be noted that the parts used in each example are based on weight.

[0072]

#### Application Example 1

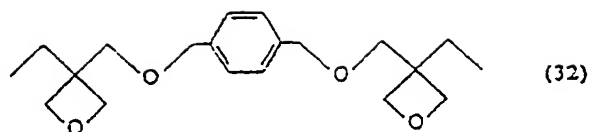
##### • Production of composition

Mixing was carried out for 100 parts of the compound having 2 oxetane rings shown below (32) (hereinafter, component A) as the compound having an oxetane ring and 4 parts of compound (33) shown below as the photocationic polymerization initiator (hereinafter, component G) to produce an energy-beam-curable composition to be used for the primary coating of optical fibers.



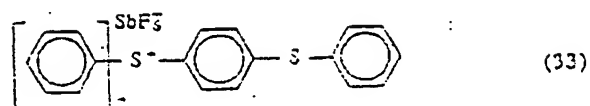
[0073]

[Structure 32]



[0074]

[Structure 33]



[0075]

For the composition and cured film produced, evaluations described below were carried out, and the results obtained are shown in Table II below.

[0075]

- Evaluation

- Curing properties

The composition produced was coated onto several glass sheets using a bar coater to form a coated film thickness of 10  $\mu\text{m}$  to produce multiple test sheets. Ultraviolet was applied to each test piece at a different level of exposure from a distance of 10 cm using a parallel beam, high-pressure mercury lamp at 80 W/cm. The exposure at which the viscosity of the coated composition is lost was defined as the minimum ultraviolet exposure ( $\text{J}/\text{cm}^2$ ).

[0077]

The composition was coated onto a glass sheet using an applicator to form a coated film thickness of 250  $\mu\text{m}$ , and ultraviolet was applied using the above-mentioned device until the viscosity was lost and a cured film was produced. The cured film was then removed from the glass sheet, and an evaluation was carried out for the tensile modulus of elasticity, heat resistance, and resistance to hydrolysis and water absorption using the methods described below.

[0078]

## ○ Tensile modulus of elasticity

Measurement of the tensile modulus of elasticity ( $\text{kgf/cm}^2$ ) /10  
for the cured film was carried out according to the method  
specified in JIS K 7113.

[0079]

## ○ Heat resistance

The cured film was stored in air at 120°C for 3 days and the  
tensile modulus of elasticity was measured as described above.  
The measured value obtained was divided by the value before the  
test. Heat resistance is excellent when the above-mentioned value  
is high.

[0080]

## ○ Resistance to hydrolysis

The cured film was stored in 80°C water for 30 days, and the  
tensile modulus of elasticity was measured as described above.  
The measured value obtained was divided by the value before the  
test. Resistance to hydrolysis is excellent when the above-  
mentioned value is high.

[0081]

## ○ Water absorption

For the cured film produced, the water absorption (%) was measured according to the method specified in JIS K 7209 (method B). Water absorption is excellent when the water absorption value is low.

[0082]

Application Examples 2-7

Components and compositions shown in Table I were used, and energy-beam-curable compositions used as primary coatings for optical fibers were produced as in the case of Application Example 1. For the compositions and cured films produced, evaluations were carried out as in Application Example 1, and the results obtained are shown in Table II.

[0083]

Comparative Examples 1-3

Components and the compositions shown in Table I were used, and energy-beam-curable compositions used as primary coatings for optical fibers were produced as in Application Example 1. For the compositions and cured films produced, evaluations were carried out as in Application Example 1, and the results obtained are shown in Table II.

[0084]

Table I

|         | A   | B  | C  | D  | E   | F  | G | H |
|---------|-----|----|----|----|-----|----|---|---|
| ① 实施例 1 | 100 |    |    |    |     |    | 4 |   |
| 实施例 2   | 50  | 50 |    |    |     |    | 4 |   |
| 实施例 3   |     |    | 25 |    | 75  |    | 4 |   |
| 实施例 4   | 75  |    |    |    |     | 25 | 3 | 1 |
| 实施例 5   | 25  |    |    | 50 |     | 25 | 3 | 1 |
| 实施例 6   | 50  |    |    |    | 25  | 25 | 3 | 1 |
| 实施例 7   | 75  |    |    | 25 |     |    | 4 |   |
| ② 比较例 1 |     |    |    |    | 100 |    | 4 |   |
| 比较例 2   |     |    |    | 75 | 25  |    | 4 |   |
| 比较例 3   |     |    |    | 25 |     | 75 | 1 | 3 |

Key: 1 Application Example  
 2 Comparative Example

[0085]

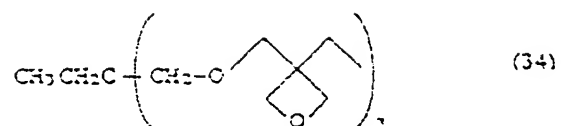
In Table I, the numbers represent parts [by weight].  
 Furthermore, in Table I, components B-H are the compounds shown below.

[0086]

• Component B (compound having 3 oxetane rings shown in [general] formula (34) below)

[0087]

[Structure 34]

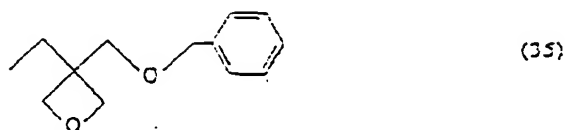


[0088]

• Component C (compound having 1 oxetane ring shown in [general] formula (35) below)

[0089]

[Structure 35]

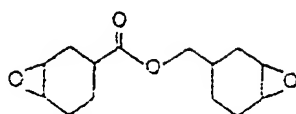


[0090]

• Component D (compound having 2 epoxy groups shown in [general] formula (36) below)

[0091]

[Structure 36]



(36)

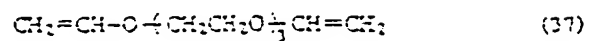
[0092]

• Component E (compound having 2 vinyl ether groups shown in [general] formula (37) below)

[0093]

[Structure 37]

/11

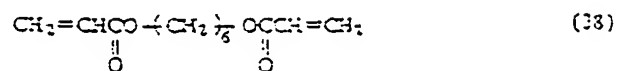


[0094]

• Component F (compound having 2 acryloyl groups shown in [general] formula (38) below)

[0095]

[Structure 38]

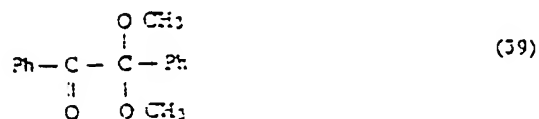


[0096]

Component H (the photoradical polymerization initiator shown in [general] formula (39) below)

[0097]

[Structure 39]





[0098]

Table II

|      | 最少紫外<br>照射量 <sup>1)</sup> | 引張弾<br>性率 <sup>2)</sup> | 耐熱性 <sup>3)</sup> | 耐加水<br>分解性 <sup>4)</sup> | 吸水率 <sup>5)</sup><br>(%) |
|------|---------------------------|-------------------------|-------------------|--------------------------|--------------------------|
| 実例1  | 0.70                      | 29                      | 0.7               | 0.7                      | 1.1                      |
| 実例2  | 0.46                      | 17                      | 0.7               | 0.8                      | 0.9                      |
| 実例3  | 0.31                      | 31                      | 0.6               | 0.6                      | 3.1                      |
| 実例4  | 0.68                      | 33                      | 0.7               | 0.7                      | 1.5                      |
| 実例5  | 0.23                      | 21                      | 0.7               | 0.5                      | 1.1                      |
| 実例6  | 0.29                      | 29                      | 0.7               | 0.6                      | 1.2                      |
| 実例7  | 0.16                      | 15                      | 0.7               | 0.6                      | 2.0                      |
| 比較例1 | 0.31                      | 20                      | 0.3               | 0.3                      | 5.6                      |
| 比較例2 | 未硬化 <sup>6)</sup>         | -                       | -                 | -                        | -                        |
| 比較例3 | 1.53                      | 8                       | 0.2               | 0.3                      | 3.4                      |

1) Units: J/cm<sup>2</sup>2) Unit: kgf/cm<sup>2</sup>

Key: 1 Minimum ultraviolet exposure<sup>1)</sup>  
 2 Tensile modulus of elasticity<sup>2)</sup>  
 3 Heat resistance  
 4 Resistance to hydrolysis  
 5 Water absorption (%)  
 6 Application Example  
 7 Comparative Example  
 8 Noncuring

[0099]

#### Effect of the invention

The curing rate is high in the energy-beam-curable composition used as a primary coating for optical fibers of the present invention, and the cured film exhibits excellent heat resistance, resistance to hydrolysis, and resistance to water absorption, as well as a low tensile modulus of elasticity, and the material produced has a very high applicability.

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